Environmental Technology Center

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CIBA–GEIGY

August 26, 1991

REC'D 8-29-91
F.B.

Mr. Frank Battaglia
USEPA Region I
Waste Management Bldg.
90 Canal St.
Boston, MA 02114

OK PER
DEB SZARRO
EPA LAB (REG I)

Dear Mr. Battaglia

Enclosed is the audit report from Region V for Savannah Laboratories which was conducted in 1990 specifically for Appendix IX. This report was previously requested to be sent from David Payne (Region V) to Deb Szarro as interagency information. Savannah Laboratories obtained permission from Gerharty and Miller to release the report to me also. Since David was in the midst of meeting several deadlines, and may not be able to call Deb for awhile, I am enclosing a copy of the audit report to assist in expediting Region I's review. This report was faxed to you 8/23 and was sent to Joanna Hall at Alliance by Federal Express (since our fax machine stopped transmitting after your report was sent).

Please review the information presented and comment by August 29th (if at all possible), so the fieldwork can remain on schedule. I look forward to your response.

Sincerely,

Diana Baldi

Diana Baldi, Administrator
National Services Contracts

Attachments

cc: Diane Leber
Ken Dupuis
Frank Saksa
Mark Houlday, Woodward Clyde
Joanna Hall, Alliance
Peter Reynolds, Woodward Clyde

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
230 SOUTH DEARBORN ST.
CHICAGO, ILLINOIS 60604

SEP 28 1990

MEMORANDUM

REPLY TO ATTENTION OF: 5SCRL

DATE:

SUBJECT: Laboratory Evaluation - Savannah Laboratories and Environmental Services, Inc., Savannah, Georgia for Portsmouth Gaseous Diffusion Plant, Portsmouth, OH., RFI

FROM: Charles T. Elly, Acting Director Chuck E Elly
Central Regional Laboratory

TO: William E. Muno, Chief
RCRA Enforcement Branch

ATTN: James Saric

I. SUMMARY AND RECOMMENDATIONS

A. Savannah Laboratories, Savannah, Georgia, was evaluated for Appendix IX groundwater analysis and Target Compound List (TCL) organic and Target Analyte List (TAL) inorganic determinations of soil/sediment for the subject RCRA Facility Investigation (RFI). The Savannah laboratory was found acceptable in capability for these determinations, but in need of completion of certain items prior to final approval for the project. The laboratory demonstrated acceptable instrument calibration for most Appendix IX contaminants in a thoughtful professional manner. The laboratory is to be complimented for their efforts. The laboratory will be doing all required analyses for the project, except the Corporation's Tallahassee, Florida Facility will be doing Method 8140 determinations of eight phosphorus containing pesticides and will serve as a back-up for volatile analyses by Method 8240 (both TCL and Appendix IX). The Tallahassee facility is a subject of another memo report.

B. The laboratory evaluation was done with no available detailed Quality Assurance Project Plan (QAPP), so that this memorandum report will hopefully provide direction for completion of a QAPP. The evaluation did reveal that the laboratory had selected test procedures appropriately for each of the Appendix IX contaminants. A QAPP for the subject RFI needs to define the contaminants to be measured by each test procedure.

C. The Savannah facility of the Savannah Laboratories was found acceptable for:

1. Sulfide analysis of waters (Appendix IX)

The in-house methodology is excellent, but is not readily referenced to standard SW-846 test procedures in a future QAPP.

2. Appendix IX and TAL metals analysis of soils and waters. One corrective action item is noted below, but overall capability is acceptable.

3. Appendix IX and TCL analyses by Method 8270.

The laboratory has a well established TCL methodology. Separate continuing calibration standards contain the extra Appendix IX compounds. Analytical equipment and instrumentation is sufficient for the large subject RFI with management of the workload. A finalized QAPP should note the non-detectability of any compounds (ex-hexachlorophene, N-nitrosodimethylamine) or elevated reporting limits due to poor instrument response. The laboratory should not waste time trying to establish acceptable response for aramite or hexachlorophene. The laboratory still has to establish their I.D. files for:

a) Chlorobenzilate
b) 2-methyl-pyridine
c) o,o,o-triethylphosphorothiate
d) 1,4 dioxane

These are contained in their standard solutions. Aramite is available as a pure compound, but has not been added to the Appendix IX calibration standard.

Recommendation: Consider the basic Method 8270 test procedure to be acceptable. Provide copies of a typical ID File, Quant Report, and Calibration File to the Central Regional Laboratory for Method 8270 when all details have been completed.

D. Method 8240 Appendix IX and TCL Volatiles.

The instrumentation (including data system) available for Appendix IX volatile analyses of groundwaters at time of the laboratory evaluation was inadequate for the large numbers of samples expected from the subject RFI. The single available instrument system was set-up to demonstrate that the laboratory could perform Appendix IX volatiles. TCL volatile analyses on 2 different instrument systems were found acceptable. Volatile analyses at the subject site are

complicated by the gross trichlorethene (TCE) concentrations in some samples, which had previously caused severe instrument downtimes from TCE contamination. The laboratory knows what it is doing, but must implement the following steps:

1. Convert the RTE-A Series instruments to Appendix IX capability, with or without capillary columns, or obtain additional instrumentation and/or data systems.

2. Implement sample screening procedures to guard against gross TCE contamination.

3. Update Appendix IX calibration standards to include isobutanol. Assure that both cis-and trans-1,2-dichloroethene are included in final TCL and Appendix IX measurements.

4. Add site-specific Freon 113 to both TCl and Appendix IX calibration standards.

5. Within the QAPP mechanism, establish policies on what to do for Appendix IX contaminant measurements in the presence of gross TCE concentrations. Do not expect to measure 2 ug/l vinyl chloride in the presence of 10 mg/l TCE, unless a separate methodology is established for vinyl chloride.

6. The measurement of vinyl chloride in the presence of high TCE concentrations should be a separate topic or analysis procedure in the QAPP to be written. Method 8010 may be appropriate for this task and is discussed later in this report.

7. When Appendix IX and TCL calibrations are completed on new GC/MS instrumentation, Savannah Laboratories should provide the Central Regional Laboratory finalized I.D. Files, and a typical calibration file and Quant Report for these contaminants.

E. Corrective Actions to the laboratory evaluation are listed below:

1. Method 8280 (Dioxins/Dibenzofurens)

   a. The signal to noise ratio specification for Mass 320 of 2,3,7,8-TCDD should be measured and verified as to acceptability.

   b. The peak resolution of native 1,2,3,4 TCDD and 2,3,7,8-TCDD should be used instead of the "labeled" isomers.

2. Method 8080 (Pesticides/PCB's)

   a. Implement an SOP with more rigorous sample extract clean-up steps as appropriate. Utilize florisil clean-ups instead of the alumina now in place. Details are listed

in the attached report. Recommendations concerning capillary columns and alpha and gamma chlordane are optional and not mandatory. Use of any extract clean-up other than alumina dictates elimination of DBC as a surrogate.

b. Management of available instrumentation versus sample load may be critical to the large sample load from the subject RFI as Method 8080 instrumentation will also be used for Method 8150 determinations. This is a comment.

c. As a minimum additional reference, 5-point calibration curves are needed for the site-specific Aroclor 1260. This compound may be the best matrix spike compound for the study.

d. Implement the 5-point reference calibration curves required by this methodology.

3. Method 8150 (Herbicides)

a. Utilize the diazomethane reagent specified by this methodology.

b. Implement the 5-point reference calibration curves required by this methodology.

c. Implement the agreed-upon MS/MSD compounds and 2,4-DB Surrogate spike compound.

4. Cyanide (Appendix IX and TCL)

Investigate the use of the phosphate buffer reagent of Footnote 15 to Table IB of 40 CFR136 in the Auto Analyzer measurement of distilled cyanide.

5. Metals (TCL and Appendix IX)

a. Implement analytical spikes, on a sample-by-sample basis, for graphite furnace atomic absorption determinations.

F. QAPP

The final draft QAPP submitted for review should address the test procedures to be used for each contaminant. Sample extraction preparation procedures should be clearly defined for both organic and inorganic analyses. Sample extract clean-ups (for organics) should also be specified. Surrogate matrix spike compounds should be established and defined for each method, if at all possible, even if acceptance limits are advisory at this time. Matrix spike compounds of Aroclor 1260 may be appropriate for Method 8080. Matrix spike compounds for Method 8240 may be a waste of time and money when gross TCE concentrations are present. Matrix spike compounds for

vinyl chloride may well be a significant audit by Method 8010 when gross TCE concentrations are present.

G. Evidential Files/Data Packages

The final QAPP should establish policies for data packages to serve as evidential files for the project, as appropriate. Items in the attached report to add to data packages are identified as sample preparation logs (including any clean-up steps), % solids determinations, MS tuning checks (CLP Form V for DFTPP or BFB), and surrogate recoveries. Gerrahty and Miller have suggested they have specifications for CLP-like data packages that are suitable for subsequent data validation and evidential files (Level V).

Savannah Laboratories will be developing reporting limits for each Appendix IX and TCL contaminant. For organic analyses (especially the GC/MS determinations), it is requested that if a contaminant is detected as being present at less than the reporting limit, that it be identified as being present and quantified with a "J" qualifier. This will be important for samples diluted because of TCE. It is intended that no compounds be reported as "J values" if compound identity is uncertain. Reporting limits are estimated conservative values and do not reflect daily instrument performance or the uncertainties in many of the water soluble volatile measurements.

cc: G. Schupp, QAS
D. Payne, CRL

ROUTING AND TRANSMITTAL SLIP Date 10-2-90

TO: (Name, office symbol, room number, building, Agency/Post)	Initials	Date
1. Jeannette Long, Vice Pres.		
2. Savannah Laboratories & Env. Services, Inc.		
3. P.O. Box 13548		
4. Savannah, GA 31416		
5.		

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As Requested	For Correction	Prepare Reply
Circulate	For Your Information	See Me
Comment	Investigate	Signature
Coordination	Justify	

REMARKS

Here are 2 copies of lab anal. reports previously sent to our Waste Management Div.

DO NOT use this form as a RECORD of approvals, concurrences, disposals, clearances, and similar actions

FROM: (Name, org. symbol, Agency/Post) David A. Payne, CKL, Log in V, EPA (5SCRL) | Room No.—Bldg. | Phone No. (312) 886-1970

5041-102

OPTIONAL FORM 41 (Rev. 7-76)
Prescribed by GSA
FPMR (41 CFR) 101-11.206

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WESTON MANAGERS DESIGNERS/CONSULTANTS
c/o U.S. EPA
ESAT PROJECT
ROOM 871
536 S. CLARK STREET
CHICAGO, IL 60605

Environmental Services Assistance Team
U. S. EPA - Region V

RCRA Facility Investigation
Portsmouth Gaseous Diffusion Plant
Laboratory On-Site Audit Report

For

Savannah Laboratories and
Environmental Services Inc./Savannah Laboratory

Submitted to:

Dave Payne
U.S. EPA

Submitted by:

Kelly D. Head
D. Kirk Cromer
ESAT

September 27, 1990
TID #05-90-06-31 (05-90-08-26)
Task # 3074 (3300)

Introduction

On July 23 through July 25, 1990, David Payne, Central Regional Laboratory (CRL), Kirk Cromer (Weston ESAT), and Kelly Head (Weston ESAT), evaluated Savannah Laboratory, Savannah, Georgia for chemical analyses to be done in support of the RCRA Facility Investigation (RFI) at the Portsmouth Gaseous Diffusion Plant, Piketon, Ohio. Analyses to be done are Target Compound List (TCL) organics for soils/solids, Target Analyte List (TAL) inorganics for soils/solids, and the full Appendix IX list compounds for ground waters. The groundwaters are also to be analyzed for Vinyl Chloride by SW-846 Method 8010.

There are several site specific contaminants which have been detected at the Portsmouth Site. These compounds are: Cr, Cr(VI), trichloroethylene (TCE), trichloroethylene metabolites, Aroclor 1260, 1,1,2-trichloro-1,2,2-trifluoroethylene (Freon 113), and other freons. Trichloroethylene has been measured in sampling wells at concentrations as high as 700 mg/l. The TCL, GC, and Appendix IX volatile analyses for the RFI will be drastically affected by the high concentration of this contaminant. A screening method was discussed with Savannah Laboratory staff. It was recommended that this method be brought on-line as soon as possible.

I. Sample Bottle Preparation

Sample bottles are to be provided by Savannah Laboratories from their Savannah facility. The laboratory cleans and prepares its bottles internally. QC samples are run from each lot and this information is available on request. They also prepare the sample bottles with the appropriate preservatives for each analysis type and color code the bottles and caps as they pack them for shipment. The entire sample bottle preparation, shipping and chain-of-custody is a very well thought out and organized procedure.

The sample holding times specified for extraction and analysis are all in conformance with project and method requirements.

II. Acid Base Netrals (Appendix IX and CLP Target Compound List)

Savannah Laboratory is acceptable and appropriate for the determination of Appendix IX semi-volatiles for water and Target Compound List (TCL) semi-volatiles for soils by SW-846 Method 8270.

The QC for the semi-volatile TCL and Appendix IX analyses will follow the CLP SOW. A five-point calibration curve is used for the TCL and Appendix IX semi-volatile compounds; a TCL calibration verification standard will be analyzed every 12 hours and the Appendix IX calibration curve will be verified every 24 hours, If not acceptable, the Appendix IX verification standard will be analyzed every 12 hours.

The Appendix IX calibration does not have the Calibration Check Compounds (CCCs) and System Performance Check Compounds (SPCCs) to follow for acceptable calibration criteria. The laboratory will use common sense in determining whether or not a new Appendix IX initial calibration is needed (RF > 0.05, %D < 40% for majority of the compounds). The initial calibration will be used for reference only.

Several of the Appendix IX semi-volatile compounds reported poor performance for SW-846 Method-8270.

Hexachlorophene low response
N-nitrosodimethylamine No response
Aramite No response

The responses of the compounds as explained below:

The hexachlorophene poor performance, is commonly observed in other laboratories. Poor separation of N-nitrosodimethylamine from the solvent phase is a typical problem, which cancels little if any response for this compound. Savannah Laboratory does have a standard for Aramite, but did not have it in the working standards.

Savannah Laboratory will be adding 1,4-dioxane to the semi-volatile portion of analysis. The lab tried to analyze this compound by purge and trap and found the response factors to be very low.

Chlorobenzilate, 2-methyl-pyridine, and 0,0,0-triethyl phosphorothioate are missing from the working ID files. However, these compounds are present in the working standards (figure 1-6). The lab evaluation reviewed the calibration procedures and found them acceptable. The poor response compounds should not hold up this RFI project.

The laboratory is following SW-846 Method 3510 (Separatory Funnel Liquid Extraction) for all extractable water samples and SW-846 Method 3550 (Sonication Extraction) for all extractable soil samples. These methodologies are acceptable for the Appendix IX compounds being determined in water and for the TCL compounds being determined in soil. The laboratories implementation of these methods also appears acceptable.

Sample clean-up procedures utilized by the laboratory are potentially inadequate, should any of the samples from this project have exceptionally high background interferences. The laboratory does not currently have a GPC for clean-up of "dirty" semi-volatile samples. If dirty samples are encountered, the laboratory routinely dilutes the extracts. Sample clean-up by dilution is not an acceptable technique due to the problem of raising the achievable detection limits. The lab has not experienced any problems with dirty samples. A GPC system is on order.

Savannah Laboratory does have sufficient instrumentation for the semi-volatile portion of analyses. Two HP-5970 GC/MS systems will be used. There is one HP-RTE-A series computer shared by the two instruments. Both systems are equipped with HP fused silica capillary columns.

The laboratory is currently experiencing a problem with the semi-volatiles; the HP fused silica capillary columns appear to degrade at a faster rate during the Appendix IX analysis (calibration) than other analyses due to the number of compounds in the standards and the reactivity of the compounds. This may cause a problem with instrument down time. This is the reason the lab would like to verify the Appendix IX semi-volatile calibrations every 24 hours in place of every 12 hours. A tabulation of the semi-volatile TCL compounds and additional Appendix IX list and chromatogram of the Appendix IX compounds can be found in the Appendix (Table 1 & 2 respectively).

III. Volatiles by GC/MS (Appendix IX and CLP Target Compound List)

The volatile analysis for TCL and Appendix IX compounds will follow SW 846 Method 8240. The QC will follow the CLP SOW. This laboratory is equipped with 3 GC/MS instruments which are set-up for VOA analysis. Two of these instruments have packed columns for TCL analysis. The third is equipped with a wide bore capillary column for the Appendix IX analysis.

The TCL analysis for soils/solids is acceptable to Region V. Savannah Laboratory does possess the additional Appendix IX compound standards. All of these compounds can be placed in one aliquot for purging. At the time of the audit the Appendix IX analysis was being performed on a GC/MS instrument with a Chem-station data system which is very slow for processing data. The ID file on the Chem station is set-up for all of the Appendix IX VOA compounds, except for Isobutyl alcohol, the instrument was not calibrated for Appendix IX analysis at the time of the evaluation. This instrument set-up would be unable to handle the number of samples expected for this project. This analysis should be done on their RTE-A series instruments. The laboratory does have a five point calibration for the Appendix IX and TCL Compounds on the Chem-station system. This instrument is currently set-up with a Restek 60m megabore capillary column which provides separation of the cis and Trans-1,2-Dichloroethane, good overall resolution and a shorter analysis time. The Savannah Laboratory is waiting for delivery of additional Restek 60m megabore capillary columns to convert the A series instruments. Once the columns have arrived, it should take about 2 weeks for the laboratory to bring the RTE-A instruments on-line with capillary columns for the volatile analysis. They will have to calibrate the instruments, create ID files, and update the ID files before analyzing any samples. At the time of the audit the columns had not been ordered.

Recommendations:

The Portsmouth Site have significant levels of trichloroethylene, dichloroethylene, 1,1,1-trichloroethane, and 1,1,2-trichloro-1,1,2-trifluoroethane (Freon 113). Because of the presence of these compounds, the samples should be screened by GC/FID prior to GC/MS analysis. The trichloroethylene has been detected at mg/l levels in previous studies for this site. At these levels, the laboratory should be very cautious and screen all samples. Geraghty and Miller, Inc. has stated they will flag the potentially high concentration samples. The Savannah Laboratory has a GC/FID available for screening, but the instrument was not operational at the time of the on-site audit. This screening GC should be set-up, calibrated, and brought into routine operation as soon as possible.

The Freon 113 was not present in the ID file or the standard mixture. It is recommended this compound be added to the target list.

A table from the Work Plan has been included to show the high levels of TCE which have previously been detected in some of the existing wells. These high levels should be taken into account for MS/MSD QC audits.

The lab should provide documentation to Region V for calibrations on the RTE-A series systems and screening method by GC/FID before approval of laboratory for this project to show they are ready for the VOA Appendix IX analysis.

The TCL and Appendix IX compounds are listed in Tables 4 and 5, respectively.

Due to the high TCE level in certain samples Appendix IX volatile the detection limits will be raised drastically. We can either delete the analysis of Appendix IX volatiles or accept elevated detection limits. This should be discussed further in future QAPP's for this site.

IV. Polychlorinated Dibenzo-p-dioxins and Polychlorinated dibenzofurans (Appendix IX)

The laboratory is required to analyze for polychlorinated dibenzo-P-dioxins (PCDDs), and polychlorinated dibenzofurans (PCDFs) (tetra through hexa isomers) by the Appendix IX list for water samples. The lab has one instrument set up for this analysis.

The dioxin/furan samples will be analyzed on a HP Chem-station system. The laboratory is equipped with a regulated access facility for the preparation of these samples. The SW-846 Method 8280 is the procedure that is to be followed for this analysis. This method is a high resolution capillary column gas chromatography/low resolution mass spectrometry technique. Isotopic dilution is the method of quantitation. The $^{13}C_{12}$, 2,3,7,8-tetrachlorodibenzo-P-dioxin isotope is used as the internal standard and $^{13}C_6$ 1,2,3,4-tetrachlorodibenzo-P-dioxin is used as the recovery standard.

A 30m capillary column is used to achieve isomer separation. A five-point calibration is used to verify the linearity of the instrument. A window solution is also used to determine the retention time window for tetra-, penta-, and hexa-isomers for dioxins and furans. The calibration and window solution are acceptable and appropriate for this RFI.

Savannah Laboratory may have some difficulty in meeting sample analysis hold times with a large number of samples due to the limitation of the one instrument.

Recommendations:

A minimum signal-to-noise S/N ratio of 50:1 for mass 320 of 2,3,7,8-TCDD is needed to verify the method sensitivity as specified in SW-846 Method 8280. The lab should include these data in their data packages.

The peak resolution between 1,2,3,4-TCDD and 2,3,7,8-TCDD must have a valley of $\leq$ 25% between peaks, in order to resolve the 2,3,7,8 isomer from the other isomers. The laboratory is using ^{13}C isomers to show the resolution. Using ^{13}C isomers does not prove the separation of the native isomers but only separation of isotopically labelled isomers. The separation of the native 1,2,3,4-TCDD and 2,3,7,8-TCDD must be supplied.

The laboratory must provide daily S/N and peak resolution data before samples can be analyzed.

V. Chlorinated Pesticides/PCBs (Appendix IX and CLP Target Compound List)

Chlorinated Pesticide/PCBs analysis will follow SW-846 Method 8080 for TCL and Appendix IX compounds. The QC will also follow this method, using the matrix spike compounds from the CLP SOW. It was recommended Aroclor 1260 be added as a matrix spike compound because of its presence at the Portsmouth Site. Savannah Laboratory is currently using dibutylchlorendate (DBC) as the surrogate standard. A five point curve for single component pesticides is used to calibrate the instrument. The multi-component analytes will be calibrated by a single point.

Available instrumentation dedicated to these analyses is acceptable. The laboratory routinely analyzes pesticides and herbicides on 2 Varian 3700 GCs equipped with dual mega-bore capillary columns, auto-samplers and data systems. A third Varian 3700 GC equipped with dual packed columns, auto-sampler and data system is routinely used for PCB analysis.

It is recommended that PCBs be analyzed on the capillary column equipment, if possible, as this would improve the quality of the analysis and eliminate analytical problems observed in some of the example data provided for the audit.

The potential exists for overloading the capacity of this equipment, depending on the rate of sample collection from the four quadrants at Portsmouth and the work load for these analyses from other clients of Savannah Laboratory. The RPM should discuss this potential problem with G&M and Savannah Laboratory to ensure sample collection is coordinated with the capability of the laboratory to accept the samples and meet required holding times.

The laboratory is following SW-846 Method 3510 (Separatory Funnel Liquid Extraction) for all water samples and SW-846 Method 3550 (Sonication Extraction) for all soil samples. These methodologies are acceptable for the Appendix IX compounds being determined in water and for the TCL compounds being determined in soil. The laboratory's implementation of these methods also appears acceptable.

Sample clean-up procedures utilized by the laboratory are potentially inadequate, should any of the samples from this project have exceptionally high background interferences. The laboratories standard cleanup procedure consists of alumina column chromatography and, in the case of suspected sulfur contamination, dilution. Sulfuric acid clean-up for PCB analysis is used by the lab. The GC analyst makes the initial decision on whether a sample requires clean-up based on the color of the extract. A Gel Permeation chromatograph was indicated to be on order for future use as a clean-up procedure.

Recommendations:

A more rigorous SOP outlining the decision process for clean-up requirements should be written.

The DBC surrogate should be replaced with 2,3,5,6-tetrachloro-m-xylene (TCMX) and decachlorobipenyl (DCB). DCB is inappropriate for packed columns.

A five point calibration should be generated for Aroclor 1260. A minimum of five peaks should be used to quantitate Aroclor 1260.

If any of the multi-component pesticides are detected, a five point calibration should be prepared, and the sample should be reanalyzed.

Alpha- and Gamma-Chlordane should be added to the Appendix IX list of chlorinated pesticides. This is an optional recommendation since the two compounds are part of the laboratory's present calibration standard.

Savannah Laboratory is not checking the DDT/Endrin breakdown on a regular basis. It is recommended this determination become part of the routine procedure.

Replace alumina column clean-up with florisil column clean-up, as endrin aldehyde, an Appendix IX analyte, is not recovered from alumina clean-up. Also, Florisil clean-ups are more effective than alumina.

Sulfur clean-up should be done by SW-846 Method 3660, not by dilution. The dilutions could raise the achievable detection

limits to an unacceptably high level. It should be noted, however, that some pesticides may be lost through this clean-up procedure.

It may be necessary to perform silica gel column chromatography on samples containing PCB's in order to meet detection limit requirements for pesticides that could be co-eluting with the PCB. A list of TCL and Appendix IX chlorinated Pesticides and PCBs is incorporated in the tables attached to this report.

VI. Chlorinated Herbicides (Appendix IX)

Savannah Laboratory is using SW-846 Method 8150 for the analysis of the chlorinated Herbicides of the Appendix IX list in waters. No herbicide analysis is required for soil/solid samples. The water extract clean-up procedure is a acid-base technique which is an integral part of the method. If soils/solids are to be analyzed, different clean-up procedures may be necessary.

The laboratory does have standards for the three herbicides of interest. SW-846 Method 8150 requires a minimum of a five point calibration curve. The Savannah Laboratory SOP SG65 for analysis of chlorinated herbicides does not specify any calibration. A five point curve is requested by the Agency.

The BF_3 method is currently in use to esterify the herbicides. This method has proven to be less efficient than the diazomethane method. Special care should be taken when working with the diazomethane method. The lab has the expertise and equipment to perform this method of esterification safely.

2,4-DB is to be used as the surrogate spike and 2,4-D, 2,4,5-T, and 2,4,5-TP as the matrix spike (MS) compounds. This QC is acceptable to Region V.

The lab uses laboratory Control Standards (LCS) to verify the sample preparation and esterification, therefore Savannah Laboratory is not required to process the working standards through the esterification procedure to create the esters. It is acceptable for Savannah Laboratory to use purchased esterified standards.

The following weaknesses in the herbicide data packages were noted:

1) the surrogate standard was not reported on any of the data summary forms.

2) sample weights were not present in any of the packages, but were located in laboratory notebooks.

Recommendations:

The BF_3 method of esterification should be replaced with the diazomethane procedure which is in SW-846 Method 8150.

Report the surrogate recovery data on their final data summary reports as part of the QC portion of the deliverables.

Sample volumes and weights must be included in the data packages, and evidential files. This would save reviewers, auditors, and Savannah Laboratory time when the data are validated by the user.

The Herbicide analyses will be conducted on the same GC/ECD instrumentation as the chlorinated Pesticide/PCB analysis. This may cause excess samples loading on these instruments and effect the timeliness of data reporting.

VII. Volatiles by GC (Vinyl Chloride)

Savannah Laboratory will also be analyzing for vinyl chloride at low levels. The procedure the lab has chosen is SW-846 Method 8010. To achieve the low detection limit of 2 ug/L capillary columns will be utilized. The primary column will be a 60m vocol wide-bore glass capillary column and a Rtx-502.2 105m glass capillary column will be used as the confirmation column. A 25 ml purge volume will be used to lower the detection limit.

A Hall detector and Flame ionization detector (FID) will be used in parallel. The range of the Hall detector is 1-20 ug/L and the FID range is 1-120 ug/L. These working ranges are acceptable to Region V. A five-point calibration curve will be used to determine the linearity and to quantify unknowns. A single point calibration check will be used to verify the calibration.

The chromatography of the primary column is acceptable. However, the 105m confirmatory column in use during the on-site evaluation appeared to have some problems; the chromatography was not as good as the 60m primary system. Corrective action was being taken by the laboratory.

The high levels of trichloroethylene (TCE) will present a difficulty for a 2 ug/L detection limit. Savannah Laboratories will have to find some way to work around high levels of TCE.

A suggestion has been made to Savannah Laboratories to shorten the purge time. With a shorter purge time of 1-2 minutes, maybe all of the vinyl chloride would purge and a minimum of TCE would be carried through the system.

VIII. Sulfide (Appendix IX)

Savannah Laboratories has developed their "in-house" method for sulfide which is the best technology reviewed so far by Region V for Appendix IX laboratories. The methodology is labeled "in-house", because many of the details are unique to the Savannah Laboratories' Apostrophy operations, but are well thought-out and superior in concept to the general outlines of "Standard Methods" and "SW-846". 'SW-846", 3rd edition leaves something to be desired for sulfide analysis.

For water samples, sulfide is precipitated with zinc hydroxide from the zinc acetate preservative. The supernatant is discarded from the precipitate, a precipitate aliquot is the re-dissolved and the resulting sulfide is determined colorimetrically by the "Standard Methods" methylene blue method. For soils/solids Savannah Laboratories has developed a distillation separation step to separate sulfide for the colorimetric determination of sulfide. For waters, Savannah Laboratories was able to demonstrate acceptable recoveries of sulfide spiked into buffered reagent water or to samples. The test procedure in effect for sulfide is acceptable and the laboratory must be complimented on their effort to develop a sound technology for this troublesome analysis. Acceptable QC procedures are in place for this analysis.

IX. Cyanide (Appendix IX and CLP Target Analyte List)

Savannah Laboratories determines cyanide by automated colorimetry using a Technicon "TrAAcs" Auto Analyzer System-(Industrial method 802-86T) after manual distillation of samples following EPA Method 335.2. Distillation of any soil samples is done in accordance with the CLP Statement of Work - Inorganics prior to the automated colorimetry.

The manual distillation step follows EPA Method 332.2 and is described acceptably in the labortory's SOP. The automated colorimetry follows the Technicon procedure but does have a deviation from EPA cyanide methodology described in EPA Method 335.2 and 40 CFR136 (Table IB). The TrAAcs system was a buffer solution of 27.2g KH_2PO_4 and 0.56g $Na_2 HPO_4$ per liter. Cyanide standard solutions for the TrAAcs are prepared using little or no sodium hydroxide content. The pH buffer system of the TrAAcs is acceptable for calibration standards to maintain a pH value less than 8.0; however manually distilled samples are collected in 1.25N sodium hydroxide (with subsequent 5 fold dilution to 0.25N sodium hydroxide content). Sample distillates are tested by the TrAAcs system with a sodium hydroxide content much greater than calibration standards. The above phosphate buffer (PH5.2) his borderline capability in maintaining a pH value of less than 8.0. Footnote 15 to Table IB of 40 CFR 136 was added by U.S. EPA

to specify a 1M KH_2PO_4 reagent for an Auto Analyzer system after manual sample distillations by Method 335.2.

Quality control for cyanide analysis is acceptably done to maintain calibration accuracy of the system. Matrix spikes of samples are only done on client request or specifications. The laboratory is performing the necessary sample pre-treatments to remove interferences.

Recommendation:

The basic cyanide methodology is acceptable in outline, but Savannah Laboratories needs to investigate the specifications of 40CFR136 for automated colorimetry. It is a good laboratory practice to maintain equivalent, pH buffer conditions for sample distillates and calibration standards. Method 335.2 requires a pH of colorimetric reaction to be less than pH 8.0 and this in why a large KH_2PO_4 buffer concentration is used for the automated system to compensate for the large caustic concentration in the manual distillate.

X. Metals by ICP Emission Spectroscopy and Graphite Furnace Atomic Absorption (Appendix IX and CLP TAL)

Metals analyses are done in an appropriate manner following CLP procedures that are equivalent to the requirements of SW-846. Sample aliquots are digested separately for the requirements of the 2 methods in accordance with the CLP. ICP emission spectroscopy (Method 6010) is done using the excellent Jarrell Ash Model ICP 61 system. Calibration and QC follow the CLP Statement of Work - Inorganics in an acceptable manner for ICP analyses. Calibration verification standards and lab Control Standards are maintained by the laboratory. Improved performance in details of these analyses were observed versus Region V's evaluation of 1988. This methodology should be considered acceptable for the subject RFI.

Two Varian Spectra 400 atomic absorption instruments, equipped with Zeeman background correction are available for graphite determinations of arsenic, lead, selenium, and thallium. Appropriate sample digestions are used in accordance with SW-846. The mandatory matrix modifiers are used for arsenic and selenium. A Jarrell Ash systems with Smith-Hieftje background correction is also used for graphite furnace determinations of lead and thallium. The Zeeman background correction is preferred or mandatory for arsenic and selenium which the laboratory uses.

Calibration and QC practices for graphite furnace atomic absorption (GFAA) determinations follow the CLP SOW except in one instance. Analytical spikes of digested sample aliquots are done at a frequency of 1 in 10, instead of every sample

aliquot. There are pros and cons for frequency of analytical spikes; however, it is Region V's policy to perform analytical spikes for every sample. If analytical spike is unacceptable (due to matrix effects), corrective actions are taken through sample aliquot dilutions or method of standard addition calculations. We do not have the knowledge whether all waters are soils from the site will demonstrate the same matrix effects.

Recommendation:

For the subject site analysis of GFAA metals, institute analytical spikes for each sample aliquot tested.

SL SAVANNAH LABORATORIES
& ENVIRONMENTAL SERVICES, INC.

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November 15, 1990

Mr. Seth Matters
GERAGHTY & MILLER, INC.
6209 Riverside Drive
Dublin, OH 43017

RE: Savannah Laboratories' Response to the EPA Region V Laboratory Evaluation to Perform Analysis at the Portsmouth Gaseous Diffusion Plant, Portsmouth, OH, RFI

Dear Mr. Matters:

We have reviewed the September 28, 1990 Summary and Recommendations from the EPA Region V audit of our Savannah, Georgia and Tallahassee, Florida laboratories. The following section contains our response to issues raised in the Summary and Recommendations.

Savannah Division Laboratory

RECOMMENDATION B. A QAPP for the subject needs to define the contaminants to be measured by each test procedure.

RESPONSE B. A QAPjP addressing the contaminants and test procedures outlined in the RCRA Facility Investigation at Piketon, Ohio was submitted on September 7, 1990.

RECOMMENDATION C-1. Sulfide analysis of waters (Appendix IX).

The in-house methodology is excellent, but is not readily referenced to standard SW-846 test procedures in a future QAPP.

RESPONSE C-1. The SL in-house sulfide procedure will be referenced in the revised QAPjP.

RECOMMENDATION C-3. Appendix IX and TCL analyses by Method 8270.

A finalized QAPP should note the non-detectability of any compounds (e.g., hexachlorophene, N-nitrosodimethylamine) or elevated reporting limits due to poor instrument response. The laboratory should not waste time trying to establish acceptable response for aramite or hexachlorophene. The laboratory still has to establish their I.D. files for Chlorobenzilate; 2-methyl-pyridine; O,O,O-Triethylphosphorothioate; and 1,4-dioxane.

RESPONSE C-3. Five-point calibrations have been completed for the TCL and Appendix IX base-neutral/acid extractable (Method 8270) constituents. As discussed in the memorandum, acceptable response has not been demonstrated for hexachlorophene due to coelution with the internal standard perylene-d12, and a suitable standard for aramite is not available. We will take the reviewer's suggestion and delete these two compounds from the reportables.

RECOMMENDATION D-1. Method 8240 Appendix IX and TCL Volatiles.

The instrumentation (including data system) available for Appendix IX volatile analyses of groundwaters at time of the laboratory evaluation was inadequate for the large numbers of samples expected from the subject RFI.

Convert the RTE-A Series instruments to Appendix IX capability, with or without capillary columns, or obtain additional instrumentation and/or data systems.

RESPONSE D-1. Two RTE-A Series instruments have been converted to capillary columns to handle the anticipated project volume. Both instruments are calibrated for Appendix IX parameters.

RECOMMENDATION D-2. Implement sample screening procedures to guard against gross TCE contamination.

RESPONSE D-2. An HP 5890 dual FID GC instrument is on-line to screen for heavily contaminated TCE and other volatile constituents.

RECOMMENDATION D-3. Update Appendix IX calibration standards to include isobutanol. Assure that both cis-and trans-1,2-dichloroethene are included in final TCL and Appendix IX measurements.

RESPONSE D-3. Isobutanol has been added to the calibration standards. Total 1,2-Dichloroethene will be included in the TCL reportables and trans-1,2-Dichloroethene in the Appendix IX reportables.

RECOMMENDATION D-4. Add site-specific Freon 113 to both TCL and Appendix IX calibration standards.

RESPONSE D-4. Freon 113 (1,1,2-Trichloro-1,1,2-Trifluoroethane) has been included in the five-point calibration standards for TCL and Appendix IX.

RECOMMENDATION D-5 and D-6. Within the QAPP mechanism, establish policies on what to do for Appendix IX contaminant measurements in the presence of gross TCE concentrations. Do not expect to measure 2 ug/l vinyl chloride in the presence of 10 mg/l TCE, unless a separate methodology is established for vinyl chloride.

RESPONSE D-5 and D-6. All Volatile samples will be screened by GC/FID prior to 8240 and 8010 analysis. The screening data will be used in determining dilutions for 8240 and 8010 based on level of sample contamination.

We agree with the reviewer's comments that standard 8010 and 8240 SW-846 methodology will not allow low detection limits for vinyl chloride in samples contaminated with high levels of TCE (or other target and/or non-target compounds). However, the only solution provided by SW-846 and CLP methodology for this dilemma is dilution prior to analysis, hence elevated PQLs. If a sample is heavily contaminated with TCE or other target and/or non-target compounds, is it necessary to detect vinyl chloride at a 2 ug/L level?

RECOMMENDATION D-7. When Appendix IX and TCL calibrations are completed on new GC/MS instrumentation, Savannah Laboratories should provide the Central Regional Laboratory finalized I.D. Files, and a typical calibration file and Quant Report for these contaminants.

RESPONSE D-7. Updated Quant Reports, Calibration files, and corresponding ID files will be submitted for all TCL and Appendix IX constituents (Method 8240) to Mr. David Payne of Region V EPA. Since a suitable standard for chloroprene cannot be obtained, we propose to eliminate this compound from the volatile reportables.

RECOMMENDATION E-1a. Method 8280 (Dioxins/Dibenzofurens)

The signal to noise ratio specification for Mass 320 of 2,3,7,8-TCDD should be measured and verified as to acceptability.

RESPONSE E-1a. Signal/Noise ratio specification of 50:1 for Mass 320 of the 2,3,7,8-TCDD isomer will be recorded to verify the method sensitivity as specified in Method 8280.

RECOMMENDATION E-1b. The peak resolution of native 1,2,3,4-TCDD and 2,3,6,8-TCDD should be used instead of the "labeled" isomers.

RESPONSE E-1b. The peak resolution of native 1,2,3,4-TCDD and 2,3,7,8-TCDD will be utilized in lieu of the "labeled" isomers to demonstrate separation of the native isomers. Daily S/N and peak resolution of the native isomers will be provided.

RECOMMENDATION E-2a. Method 8080 (Pesticides/PCB's)

Implement an SOP with more rigorous sample extract clean-up steps as appropriate. Utilize florisil clean-ups instead of the alumina now in place. Recommendations concerning capillary columns and alpha and gamma chlordane are optional and not mandatory.

RESPONSE E-2a. SL will utilize florisil column clean-up procedures for the Pesticide/PCB analysis.

Alpha and Gamma chlordane isomers will be reported in lieu of technical chlordane.

DBC (decachlorobiphenyl) and TCMX (2,3,5,6-Tetrachloro-m-xylene) will be used as surrogates for EPA Method 8080.

RECOMMENDATION E-2b. Management of available instrumentation versus sample load may be critical to the large sample load from the subject RFI as Method 8080 instrumentation will also be used for Method 8150 determinations.

RESPONSE E-2b. An additional GC equipped with dual ECD is on-line to accommodate the anticipated project volume.

RECOMMENDATION E-2c and E-2d. As a minimum additional reference, 5-point calibration curves are needed for the site-specific Aroclor 1260. This compound may be the best matrix spike compound for the study.

Implement the 5-point reference calibration curves required by this methodology.

RESPONSE E-2c and E-2d. We propose to use five-point initial calibrations for the CLP standard mix 1660 (Aroclor 1016 and 1260) and all single component pesticides except chlorobenzilate, isodrin, and kepone. A single-point initial calibration will be used for toxaphene and other aroclors, chlorobenzilate, isodrin, and kepone. Continuing calibrations will be analyzed (per 8080 methodology) for all single components except isodrin, chlorobenzilate and kepone. Continuing calibrations will not be run for chlorobenzilate, isodrin, kepone,

toxaphene and the aroclors unless these parameters are detected (per 3/90 CLP protocols).

RECOMMENDATION E-3a. Method 8150 (Herbicides)

Utilize the diazomethane reagent specified by this methodology.

RESPONSE E-3a. EPA SW-846 Method 8150 procedures for herbicide extraction, hydrolysis and solvent clean-up of the liquid matrix will be followed. We propose a modification of the esterification procedure by substitution of boron-trifluoride-methanol due to the safety hazard associated with use of the diazomethane reagent (explosive and carcinogenic). The use of BF3 as an alternate reagent for esterification for aqueous samples is documented in EPA Method 7. Our in-house data (using BF3) demonstrates acceptable recoveries for acid forms of the three Appendix IX herbicides.

RECOMMENDATION E-3b. Implement the 5-point reference calibration curves required by this methodology.

RESPONSE E-3b. Five-point calibration curves will be run for 2,4-D, 2,4,5-TP Silvex, and 2,4,5-T.

RECOMMENDATION E-3c. Implement the agreed-upon MS/MSD compounds and 2,4-DB Surrogate spike compound.

RESPONSE E-3c. DCAA (2,4-Dichlorophenylacetic Acid) and 2,4-DB will be utilized as surrogate compounds and 2,4-D, 2,4,5-TP Silvex and 2,4,5-TP will be utilized as the MS/MSD compounds.

RECOMMENDATION E-4. Cyanide (Appendix IX and TCL)

Investigate the use of the phosphate buffer reagent.

RESPONSE E-4. EPA Method 9010 will be utilized for cyanide determinations. The SL SOP for cyanide determination by autoanalyzer requires the use of a phosphate buffer with twice the capacity of that required in Method 9010. By employing distilled standards as a routine part of our QC procedures for cyanide, we have demonstrated that this buffer has adequate acid-base neutralizing capacity to accommodate the sodium hydroxide-containing distillates. Although not stated explicitly in our SOP, calibration standards are prepared in 0.25 M sodium hydroxide, thus eliminating this matrix difference in the samples and standards. A revised SOP will address this concern.

RECOMMENDATION E-5a. Metals (TCL and Appendix IX)

Implement analytical spikes, on a sample-by-sample basis, for graphite furnace atomic absorption determinations.

RESPONSE E-5a. Post-analytical spikes will be analyzed on each sample for graphite furnace determinations.

RECOMMENDATION F. The final draft QAPP submitted for review should address the test procedures to be used for each contaminant. Sample extraction preparation procedures should be clearly defined for both organic and inorganic analyses. Sample extract clean-ups (for organics) should also be specified. Surrogate matrix spike compounds should be established and defined for each method, if at all possible, even if acceptance limits are advisory at this time.

RESPONSE F. The QAPjP will be revised to list all methods, preparation and clean-up procedures which will be used on this project. Surrogates and matrix spike compounds are also listed along with acceptance criteria.

RECOMMENDATION G. Evidential Files/Data Packages

The final QAPP should establish policies for data packages to serve as evidential files for the project, as appropriate.

Savannah Laboratories will be developing reporting limits for each Appendix IX and TCL contaminant. For organic analyses (especially the GC/MS determinations), it is requested that if a contaminant is detected as being present at less than the reporting limit, that it be identified as being present and quantified with a "J" qualifier.

RESPONSE G. The QAPjP addresses deliverables which will be provided to G&M for use in the evidential files which will be purged at a periodic frequency for inclusion in the Martin Marietta Evidential files. Developing a deliverable package which could be transferred via modem or computer downloading was an important factor in designing the deliverables package.

As per Region V request, "J" values will be provided for all GC/MS volatile and semivolatile analysis. CLP 2/88 methodology for this data qualifier will be utilized.

Tallahassee Division Laboratory

RECOMMENDATION B. The laboratory evaluation was done with no available detailed Quality Assurance Project Plan (QAPP), so that this memorandum report will hopefully provide direction for completion of a RFI QAPP.

RESPONSE B. A QAPjP addressing the contaminants and test procedures outlined in the RCRA Facility Investigation at Piketon, Ohio was submitted on September 7, 1990.

RECOMMENDATION C-1a. Method 8140 analysis of waters (Appendix IX)

Define whether Method 8140 or 8141 (capillary columns) will be used.

RESPONSE C-1a. The Tallahassee Laboratory will utilize capillary columns for the determination of compounds listed in Method 8140.

RECOMMENDATION C-1b. Define the surrogate matrix spike compound (Ronnel) to be used.

RESPONSE C-1b. The surrogate matrix spike compound Ronnel will be utilized.

RECOMMENDATION C-1c. Define the matrix spike compounds to be used. The three compounds in effect are acceptable.

RESPONSE C-1c. Disulfoton, Ethyl Parathion, and Sulfotepp will be used as the matrix spike compounds for the Appendix IX 8140 constituents.

RECOMMENDATION D-1. Method 8240 Determination of Appendix IX Volatiles.

Complete the Quant I.D. File for Appendix IX volatiles to be determined.

RESPONSE D-1. Updated Quant Reports, Calibration files, and corresponding ID files will be submitted for all TCL and Appendix IX constituents (Method 8240) to Mr. David Payne of Region V EPA.

RECOMMENDATION D-2. Implement sample screening by GC/FID to minimize instrument down-time from TCE contamination.

RESPONSE D-2. All volatile samples will be screened by GC/FID prior to 8240 analysis. The screening data will be used to determine dilutions for 8240 analysis based on the level of sample contamination.

RECOMMENDATION D-3. Place Freon 113 in volatile calibration standards and ID files for both TCL and Appendix IX determinations.

SAVANNAH LABORATORIES & ENVIRONMENTAL SERVICES, INC.

RESPONSE D-3. Freon 113 (1,1,2-Trichloro-1,1,2-Trifluoroethane) has been included in the five-point calibration standards for TCL and Appendix IX.

RECOMMENDATION E. Comparability of Volatile Data Between Tallahassee and Savannah Facilities.

A finalized QAPP should define whether the dichlorobenzenes are to be done by 8240 or 8270. Comparability of compound identifications is not a problem between the two laboratories.

RESPONSE E. Dichlorobenzenes will be analyzed using Method 8270 as referenced in the project specific QAPjP submitted to EPA Region V.

I hope our responses to your recommendations have been satisfactorily addressed. If our responses are acceptable, we will include them in a revised QAPjP.

We appreciate the effort spent by the EPA Region V audit team and the G&M project manager team in assisting our staff at Savannah Laboratories an Environmental Services, Inc. to comply with the audit's recommendations and comments.

Sincerely,

Janette D. Long

Janette D. Long
Project Manager

JDL/pat